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5 FIBERS – SYNTHETIC

5.1 Analytical Approach

- 5.1.1 List and describe each textile item. Include the label information as to fiber content, brand or manufacturer, manufacturer's numbers such as the RN# or WPL# and the size. Include a brief description of any design, logos or lettering present. Make a notation as to whether the Item is intact or not buttons missing, apparent rips or tears, etc.
- 5.1.2 The physical, chemical and optical properties of known and/or questioned fibers are noted and recorded. Examples of these properties are as follows: Physical color (physical and instrumental using the microspectrophotometer), cross-section, diameter, delustrant, surface characteristics; Chemical microsolubility tests, microchemical tests, FTIR, PGC; Optical birefringence, sign of elongation, extinction and fluorescence. Refractive index is optional.
- 5.1.3 If a sufficient quantity of questioned fibers exists, then the known and questioned fibers will be compared and the properties noted in a side-by-side examination as follows: microchemical tests, cross-sectioning, polarized light microscopy, comparison microscopy, microspectrophotometry (MSP) and either FTIR or PGC with the order of these exams and any additional testing left to the discretion of the examiner and based upon the evidence at hand.
- 5.1.4 Synthetic fiber examiners will also identify and compare natural fibers to the extent that they are found in textile materials. These natural fiber types would include, but not be limited to, cotton, wool, ramie and flax. The exams used for these fiber types would generally mirror those for synthetic fibers with the exception of instrumental analysis.
- 5.1.5 Numerous excellent references are available to supplement the basics described herein and therefore, that material will not be duplicated here. This is particularly true for the areas of ropes and cordage, buttons, and fabric construction, in addition to identification and comparison of synthetic fibers.
- 5.1.6 Minimum Standards and Controls
 - 5.1.6.1 The comparison microscope data of all positive, probative associations will be verified by a second qualified examiner. The original fiber worksheet(s) will be initialed and dated by the second examiner in the space labeled "verification". The verification includes the K and Q comparison of those features determinable by viewing with both transmitted light and polarized light.
 - 5.1.6.2 Any mounting media with a stated expiration date will not automatically be discarded after the stated date. As long as the mounting media has not yellowed and continues to "flow" properly, as determined by the examiner, then it may continue to be used.

5.1.7 References

5.1.7.1 <u>Forensic Examination of Fibres</u>, 2nd ed., J. Robertson and M. Grieve, eds., Taylor and Francis, London, 1999.

5.2 Recovery of Hairs and/or Fibers

5.2.1 Purpose

To examine evidence to locate, recover and preserve hairs/fibers for identification and/or comparison purposes.

- 5.2.2 Summary
 - 5.2.2.1 Generally speaking, submitting the article or articles of evidence to the laboratory for the examiner to process is the best approach to the recovery of hairs and/or fibers. There are instances where this is not practical or possible, such as recovering hairs and/or fibers from wall-to-wall carpeting, a large piece of

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furniture, or a vehicle. In these instances, the recovery may be accomplished at the scene with the aid of an alternative light source, if available, and the recovered hairs and/or fibers submitted for examination.

5.2.2.2 The order of preference for the recovery of hairs and/or fibers is manual removal with forceps followed by taping with Post-It Notes. Gentle scraping may be necessary in certain instances. Vacuuming is rarely, if ever, performed because the debris recovered represents far more than recent hair and/or fiber transfers. However, hairs and/or fibers recovered with these methods, when submitted as evidence, will be examined to the best of the laboratory's ability.

5.2.3 Minimum Standards and Control

- 5.2.3.1 The examiner shall change the examination paper between victim and suspect or scene exhibits. The examiner may change the paper between multiple victim, suspect or scene items, as necessary.
- 5.2.3.2 There should be only one exhibit opened at a time, unless two separate areas exist for this purpose.
- 5.2.3.3 The examiner shall change gloves and clean their tools between examining the evidence from the victim and the evidence from the suspect.
- 5.2.3.4 If possible, the victim's evidence and suspect's evidence should be examined in separate rooms. If this is not possible, then the separation of victim and suspect evidence in time and/or space will be necessary. Document in case file notes.
- 5.2.3.5 Use separate laboratory coats and evidence collection rooms, if available, for examining materials from victim and suspect to prevent possible cross-transfer contamination.
- 5.2.3.6 Avoid drafts around the examination area.

5.2.4 Analytical Procedure

- 5.2.4.1 Spread a clean piece of paper on the examination surface.
- 5.2.4.2 Examine each item of evidence visually or with the aid of an illuminated magnifier, UV light or alternative light source.
 - 5.2.4.2.1 If the item being examined contains hairs and/or fibers that are readily visible, collect these hairs and/or fibers with forceps. As hairs and/or fibers are collected, they should be placed in glassine packets or affixed to Post-It notes.
 - 5.2.4.2.2 Take care with bulky items which require repositioning on the examination table, to avoid the loss of hairs and/or fibers in the repositioning process.
- 5.2.4.3 Post-It Notes or other light tack adhesive tapes may be used to recover hairs and/or fibers. The adhesive surface is placed on the item being examined and then pulled away. Hairs and/or fibers will adhere to the adhesive on the tape.
 - 5.2.4.3.1 This method may be especially useful on large items or dark-colored items on which hairs and fibers of interest may be difficult to see.
- 5.2.4.4 Scraping is generally discouraged as a method of collection for hairs and/or fibers. If scraping is necessary, the item to be examined is suspended above the examination surface and very gently scraped with a spatula. Scraping in a downward direction allows surface hairs and/or fibers to fall onto the examination paper for collection.

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5.3 Synthetic Fiber Identification

5.3.1 Purpose

The purpose of synthetic fiber identification is to identify man-made fibers as to their generic class.

5.3.2 Safety Considerations

5.3.2.1 The use of xylene substitute, Permount, Pro-Texx, or Meltmount to mount the synthetic fibers requires caution. The process may be carried out in a well ventilated area or by using a "Nederman" point-of-use vent, if one is available.

5.3.3 Minimum Standards and Controls

- 5.3.3.1 A reference collection of synthetic fibers mounted on glass microscope slides, as well as those not mounted, may be useful to the examiner when identifying generic class.
- 5.3.3.2 A reference collection of FTIR spectra of known synthetic fiber generic classes and sub-classes is necessary. The resolution of the known fiber spectra must be equal to or better than that of the sample.

5.3.4 Analytical Procedures

- 5.3.4.1 Observe the physical characteristics using the stereo microscope.
- 5.3.4.2 Observe the optical properties of the fibers using the polarized light microscope.
- 5.3.4.3 Refer to the table of optical properties of common fibers. (See ¶ 5.4.4.1.5)
- 5.3.4.4 Obtain an FTIR spectrum of the fiber. This is most typically accomplished using the microcompression cell with diamond windows.
- 5.3.4.5 Compare the spectrum obtained with reference spectra of synthetic fibers.
- 5.3.4.6 Determine the solubility of the fibers in chloroform, acetone, m-cresol, DMF, 75% sulfuric acid, concentrated nitric acid, concentrated hydrochloric acid, LeRosen, and others such as 15% hydrochloric acid or HFIP, as needed. (See Section 5.6) Also, note any color reactions that take place. The chemicals and reagents used are the discretion of the examiner and should be based upon the results of the other examinations.
- 5.3.4.7 Refer to the table of solubilities of common fibers. (See ¶ 5.4.4.8.6)

5.3.5 References

- 5.3.5.1 Gaudette, B.D. In *Forensic Science Handbook*; Saferstein, R., Ed.; Prentice Hall: Englewood Cliffs, N.J., 1988; Vol. II, Chapter 5.
- 5.3.5.2 Technical Working Group for Materials Analysis, Forensic Fiber Examination Guidelines, January 1998.

5.4 Synthetic Fiber Comparison

5.4.1 Purpose

To determine if fibers from different sources could have had a common origin.

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- 5.4.1.1 The comparison of questioned fibers with fibers from a known source is performed in every step of the examination once the questioned fibers are recovered and the standard fibers are collected from a known source.
- 5.4.1.2 The examiner can approach the fiber comparison by setting out to show that the samples are different. The failure to detect any significant differences, after exhausting the methodology available to the examiner, results in the conclusion that the fibers could have the same origin.
- 5.4.1.3 Generally, destructive testing is performed after all non-destructive testing is complete when sample size is limited.
- 5.4.2 Apparatus and Materials
 - 5.4.2.1 Pro-Texx (RI = 1.495 \pm 0.005), Permount (RI = 1.525), Norland Optical Adhesive 60° (RI = 1.56) or Meltmount (RI = 1.662), xylene substitute (RI = unknown)
- 5.4.3 Minimum Standards and Controls
 - 5.4.3.1 The comparison microscope data of all positive, probative associations will be verified by a second qualified examiner. The original fiber worksheet(s) will be initialed and dated by the second examiner in the space labeled "verification". The verification includes the K and Q comparison of those features determinable by viewing with both transmitted light and polarized light.
 - 5.4.3.2 Reagents shall be prepared in accordance with the QA/QC protocol (Appendix 4) and reagent reliability checks will be recorded.
 - 5.4.3.3 The known and questioned fibers shall be examined at the same time, in a side-by-side fashion. The results shall be recorded on a fiber worksheet.
 - 5.4.3.4 Extreme caution must be used when handling known and questioned fibers to avoid any possibility for cross-contamination.
 - 5.4.3.5 Use the same mounting media for known and questioned fibers.
- 5.4.4 Analytical Procedures
 - 5.4.4.1 Polarized Light Microscopy

Determination of generic type may frequently be accomplished with polarized light microscopy.

- 5.4.4.1.1 Prepare temporary or permanent mounts of the known and questioned fibers.
- 5.4.4.1.2 Observe both physical and optical properties of the fibers using either the stand-alone polarized light microscope or the comparison polarized light microscope. NOTE: If the fibers are observed with the stand-alone polarized light microscope at this time, they MUST also be compared on the comparison microscope at some time during the examination.
- 5.4.4.1.3 Using the fiber worksheet (Appendix 19), record the physical and optical properties. These will include at a minimum: color, delustrant, diameter, extinction, birefringence, sign of elongation and optical cross-section.
- 5.4.4.1.4 The refractive index may be noted. This is typically accomplished by a general comparison of the Becke line movement with the particular mounting media in use.

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5.4.4.1.5 Expected Results

Low Birefringence: Arnel (+)

Dynel (+); n's ca 1.534

Cellulose Acetate (+); n's ca 1.474

Orlon (-); n's ca 1.513

Moderate Birefringence: Viscose Rayon (+); n's = 1.53 & 1.55

Cotton - twists; (+); no extinction; n's = 1.53 & 1.58 Wool & other animals (+); scales; n's = 1.5 & 1.6 Silk; crossover marks; (+); n's = 1.54 & 1.59

Linen & other bast fibers (+); nodes; n's = 1.53 & 1.59

High Birefringence: Nylon; n's < 1.66

Dacron; $n_{\parallel} = 1.72 \& n_{\perp} = 1.53$ Nomex; $n_{\parallel} = 1.70 \& n_{\perp} = 1.67$

Kevlar; $n_{\parallel} > 2.30$

5.4.4.2 Fluorescence Microscopy

Some fibers fluoresce when exposed to different wavelengths of light. Of those fibers, the wavelength and intensity of emission under different excitation wavelengths are important. Fluorescence can be caused by optical brighteners, detergents, bleaching agents, dyes, the chemical structures or other additives.

- 5.4.4.2.1 Generally done at the same time as the observation of physical and optical properties.
- 5.4.4.2.2 Fluorescence cubes to be used are **WU** (wide UV 330 385 nm), **WBV** (wide blue violet range 400 440 nm), **WB** (wide blue range 450 480 nm) and **WG** (wide green range 510 550 nm). These filter blocks include excitation and barrier filters. Record observations on the fluorescence worksheet.
- 5.4.4.2.3 A significant difference in fluorescent properties between known and questioned fibers at any of these excitation wavelengths is cause for elimination.
- 5.4.4.2.4 Certain mounting media will fluoresce. Non-fluorescing media should be used to achieve optimum contrast with the background.
- 5.4.4.3 Comparison Microscope
 - 5.4.4.3.1 Observe known and questioned fibers in temporary or permanent mounts with the comparison microscope.
 - 5.4.4.3.2 Record match or nonmatch.
- 5.4.4.4 Microspectrophotometry
 - 5.4.4.4.1 Obtain spectra of questioned and known fibers mounted in the same media. If sample amount is sufficient, mount the fibers in the permanent mount of choice for best data.
 - 5.4.4.4.2 Range of colors, color intensity and distribution as well as cross-sectional shape should be considered when determining the number of spectra to be collected for the comparison on the known and questioned fibers.

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	5.4.4.4.3	Comparison should be performed by overlaying t major discrepancies between the two are reason f				
5.4.4.5	FTIR					
	5.4.4.5.1	Obtain spectra of the known and questioned fiber placing them on a KBr window or mirrored slide with diamond windows.				
	5.4.4.5.2	Comparison should be performed by overlaying t major discrepancies between the two are reason f sample several known or questioned fibers or to t given fiber to determine the reproducibility of the	or elimination. It may be necessary to take several spectra along the length of a			
5.4.4.6	PGC					
	5.4.4.6.1	Obtain pyrograms of the known and questioned f the gas chromatograph if there is a sufficient quant				
	5.4.4.6.2	Compare the known and questioned pyrograms b discrepancies between the two are reason for elim				

determine the reproducibility of the fibers.

5.4.4.7 Cross-sections

Frequently the cross-section of a fiber can be determined from the longitudinal view, also known as optical cross-sectioning. This may be the only technique available for cross-sections if the questioned fiber is too short.

several known or questioned fibers or to run several pyrograms of the same fiber to

- 5.4.4.7.1 Prepare cross-sections of the known and questioned fibers, treating both the known and questioned fibers in the same manner, using one of the following methods:
 - 5.4.4.7.1.1 Polyethylene sheets Single fibers are placed between two plastic sheets. The plastic "sandwich" is placed on a glass microscope slide, another slide or a cover slip is placed over the plastic "sandwich" and this is then transferred to a hot plate set on low. Some pressure may need to be applied as the plastic melts around the fiber. A new single-edged razor blade or scalpel blade may be used to slice thin cross-sections of the sandwiched fiber. The remaining portion of fiber is easily removed by cutting and lifting away the plastic sheets.
 - 5.4.4.7.1.2 Super Glue [®]Gel- Place the fibers to be cross-sectioned on a microscope slide. Add enough glue to cover the fibers. After fully drying, use a new single-edged razor blade or scalpel blade may be used to slice thin cross-sections of the fiber.
 - 5.4.4.7.1.3 Norland Optical Adhesive 60[®] Place the fibers to be cross-sectioned on a microscope slide. Add enough adhesive to cover the fibers. Expose to long wave ultraviolet light (320-400 nm) for approximately 5-10 minutes, or until completely cured. A new single-edged razor blade or scalpel blade may be used to slice thin cross-sections of the fiber.
- 5.4.4.7.2 Compare the prepared cross-sections with the comparison microscope and record observations.

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5.4.4.8 Microchemical Tests

- 5.4.4.8.1 The chemicals and reagents used are at the discretion of the examiner and should be based upon results from other examinations and sample condition and quantity.
- 5.4.4.8.2 The solubility of synthetic fibers in specific liquids allows an examiner to identify the generic class of the fiber. Observation of color reactions in the reagents allows the side-by-side comparison of the reaction of the dyes used in the known and questioned fibers.
- 5.4.4.8.3 Microchemical test reagents that are used are: chloroform, acetone, m-cresol, DMF, 75% sulfuric acid, concentrated nitric acid, concentrated hydrochloric acid, LeRosen, and others such as 15% hydrochloric acid or HFIP, as needed.
- 5.4.4.8.4 Cut small portions of the known and questioned fibers and place them in welled spot plates or on a microscope slide under a cover slip.
- 5.4.4.8.5 Add a drop or two of the microchemical test reagent. Observe and record the reaction with the aid of the stereomicroscope. In addition to notations as to soluble, partially soluble or insoluble, record any color changes that occur using the fiber worksheet.
- 5.4.4.8.6 Expected Results

Key to solubility/microchemical reactions:

- S = Soluble/Disintegrates (fades, splinters, or breaks apart and goes into solution)
- P = Partially Soluble (not all portions of a fiber are soluble within 5 minutes)
- I = Insoluble (no reaction)

Determine the generic class from the following table:

	ACT S/T	ACR	MOD D/V	NYL	NYT	OLE PE/PP	POL	RAY	SPA	CO/ FL	SI	WO
Acetone	S/P	I	P/I	I	I	I/I	I	I	I	I	I	I
Chloroform	I/S	I	I/I	I	I	- /I	I	I	I	I	I	I
m-Cresol	S/S	I	I/I	S	I	I/I	I	I	I	I	I	I
DMF	S/S	P	S/S	I	I	I/I	I	I	I	I	I	I
conc. HCl	S/P	I	I/I	S	I	I/I	I	S	I	I	I	I
conc. HNO ₃	S/S	S	I/-	S	S	I/I	I	I	P	I	P	P
75% H ₂ SO ₄	S/S	-/S	I/I	S	I	I/I	I	S	P	P	P	I
LeRosen				S		I/I	S					
15% HCl				S/I								
HFIP							S					

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ACT S/T = acetate secondary/triacetate

ACR = acrylic

MOD D/V = modacrylic dynel/verel

NYL = nylon (for 15% HCl: Nylon 6 = S; Nylon 6-6 = I)

NYT = nytril

OLE PE/PP = olefin polyethylene/polypropylene

POL = polyester

RAY = rayon

SPA = spandex

CO/FL = cotton/flax

SI = silk

WO = wool

5.4.5 References

- 5.4.5.1 Technical Working Group for Materials Analysis, Forensic Fiber Examination Guidelines, January 1998.
- 5.4.5.2 <u>Forensic Examination of Fibres</u>, 2nd ed., J. Robertson and M. Grieve, eds., Taylor and Francis, London, 1999.

5.5 Documentation

- 5.5.1 The examiner's notes will include a description of the known item (for example, coat) listing color, size, (manufacturer and fiber content, if available).
- 5.5.2 The examiner's notes will include a description of the questioned fiber(s), including color.
- 5.5.3 The fiber content of the known will be confirmed/determined.
- 5.5.4 In addition to individual case notes, all worksheets, FTIR spectra with appropriate reference spectra, MSP spectra and/or pyrograms with a GC conditions sheet will be included in the case file documentation.
- 5.5.5 FTIR spectra will be labeled as follows:
 - FS Lab #, Item # and how prepared (e.g., diamond cell)
 - Date and time
 - Filename (optional)

5.6 Report Wording

- 5.6.1 Unknown fibers that only require identification are reported as belonging to one of the following generic classes:
 - Acetate
 - Acrylic
 - Aramid
 - Modacrylic
 - Nylon
 - Olefin
 - Polyethylene
 - Polypropylene
 - Polyester
 - Rayon
 - Saran

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	:	Spandex Triacetate Vinal Vinyon		
	5.6.1.1	Color, cross-sectional shape or end use may be reported as app	ropriate.	
	For exam	ple: Item contained red, trilobal, nylon fibers that may be four	nd in, but are not lim	ited to, some carpets
5.6.2	If the kno	own and questioned fibers can be eliminated based upon any of the	e testing the report v	vill generally read:
		fibers could not be associated with the Item fibers due to d or, microscopic properties, optical properties, or chemical proper		
5.6.3	If the kno read:	own and questioned fibers cannot be eliminated based upon any or	f the testing the repo	rt will generally
	5.6.3.1	The Item and fibers matched in physical, chemical and o common origin.	ptical properties and	l could have had a
	5.6.3.2	Several of the fibers recovered from Item matched the fiber (carpet, shirt, sweater, blanket, etc.) in physical, chemical and otherse matching fibers could have had a common origin.		
	5.6.3.3	The red acrylic (polyester, nylon, etc.) fiber in Item matche (known) in (physical, chemical and optical properties). There have originated from the (known).		
5.6.4	If foreign read:	fibers were recovered and knowns are being requested for compa	arison purposes the i	report will generally
	Foreign (a known s	color and/or type) fibers were recovered from Item(s) which w source is located, resubmit Item(s) along with the known for c	vere suitable for com omparison purposes	parison purposes. If
	5.6.4.1	It may also be appropriate to include a general statement as fol	lows:	
	The fit	pers recovered from Item consisted of various colors of nat	ural and synthetic fi	bers.
5.6.5	If the exa	miner is requested to make a comparison based on fabric construc	ction the report will	generally read:
	matched i	were consistent in overall color and (knitted or weave) construction physical, chemical and optical properties. It was concluded that e same set (or single unit).		
5.6.6	instance,	miner should be able to convey information on how "common" a white (colorless) cotton or white (colorless) and "indigo" blue co d to have no evidential value due to their prevalence.		
5.6.7		y, textile materials are mass produced and it is not possible to state		

- A disclaimer statement may be used (for example, It is pointed out that fibers do not possess a sufficient number of 5.6.8 unique individual microscopic characteristics to be positively identified as having originated from a particular source to the exclusion of all others.)